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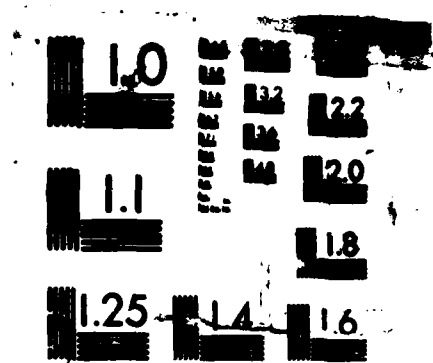
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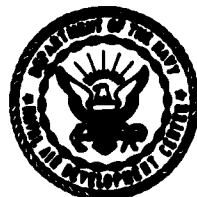




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CORROSION BEHAVIOR OF METAL MATRIX COMPOSITES

R.C. Paciej and V.S. Agarwala
Air Vehicle and Crew Systems Technology Department
NAVAL AIR DEVELOPMENT CENTER
Warminster, PA 18974

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→ Metal-matrix composites suffer from preferential corrosion because of their non-homogeneous composition and structure. In particular, aluminum alloy matrix materials are very susceptible to corrosion, when the dispersoid is a noble phase, such as SiC. This paper describes the pros and cons of what inadequate processing and fabrication can do to corrosion behavior of the composites.					
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TABLE OF CONTENTS

	Page
LIST OF TABLES	ii
LIST OF FIGURES	ii
INTRODUCTION.....	1
EXPERIMENTAL PROCEDURE.....	1
Materials.....	1
Corrosion Studies.....	3
Electrochemical Studies	3
RESULTS AND DISCUSSION	3
Corrosion Studies.....	3
Electrochemical Studies	7
CONCLUSIONS	11
REFERENCES	12

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LIST OF TABLES

Table		Page
1	Elemental Composition of Material	2
2	Heat Treatments for PM SiCp/Al MMC and Ma-87 Al Alloy	2
3	Physical Properties of the SiCp/Al MMC	2
4	Typical Electrochemical Parameters of Materials in 3.5% NaCl Solution at pH 2 ...	9
5	Typical Electrochemical Parameters of materials in 3.5% NaCl Solution at pH 6 ...	9

LIST OF FIGURES

Figure		Page
1	Effect of pH on the corrosion rates of the MMC and the MA-87 Alloy specimens with exposure time in pH 2 and 6, 3.5% NaCl	4
2	Weight-loss test specimens after exposure in pH 2 and 6, 3.5% NaCl for 3 to 10 days	5
3	Exfoliation test specimens prior to removal of corrosion products	6
4	Optical micrographs of the S-L face of the SiCp/Al alloy MMC showing: (A) and (B) Al alloy and SiCp free zones, (C) uniform SiCp distribution and (D) clustering of SiC particles	8
5	Potentiodynamic polarization behavior of the MMC and the MA-87 alloy specimens	10

INTRODUCTION

Most corrosion studies on SiC/Al alloy metal matrix composites (MMCs) have been focused on the effects of reinforcement material on their pitting potential, pit morphology and general corrosion susceptibility in NaCl solutions (1-9). The literature has been sparse on studies relating the metallurgical variables, such as fabrication and processing, with the MMC's corrosion susceptibility (5,9). The inherent processing problems of producing an extruded MMC plate has been shown to degrade the MMC's corrosion resistance in marine environments (9). One current problem is the difficulty of producing a homogeneous MMC plate (8-11). Thermal processing has been also reported to have a detrimental effect on the corrosion properties of MMCs (9). The heat treatments for SiC/Al alloy MMCs may have to be different than those conventionally used for the matrix material; since the reinforcement phase (SiC) has quite different thermal conducting properties than the Al alloy (12,13).

In a recent study, the techniques used for fabricating and processing a MMC were found to adversely affect the MMC's corrosion susceptibility (9). Corrosion and electrochemical investigations were made on a powder metallurgy (PM) SiC (Whisker)/7091Al-T6 MMC in 3.5% NaCl. It was found that the composite plate's exterior (skin areas) was preferentially corroded and its overall corrosion resistance was poor. The factors responsible for the observed corrosion behavior were as follows: (1) elemental segregation, (2) voids, (3) poor homogeneity of reinforcement phase (i.e. Al alloy and SiCw rich zones), (4) unstringered PM Al alloy material, (5) recrystallization of portions of the MMC and nonuniform plastic deformation caused by billet extrusion, and (6) residual stresses and cold-worked regions not removed by the conventional solution heat treatment. A modified solution heat treatment was employed which eliminated the preferential attack of the composite.

The purpose of this investigation was to study the effects of fabrication and processing on the corrosion susceptibility of an extruded PM SiC/Al alloy MMC plate. To produce the plate material, a MMC billet was extruded at an extrusion ratio of 20 to 1 rather than 12 to 1 used in the earlier study (9). This extrusion ratio was applied to enhance MMC homogeneity, decrease matrix porosity and the size of inclusions. Also, SiCp (p=particle) reinforcement was used instead of SiCw(w=whisker) to improve conditions for billet fabrication and extrusion.

EXPERIMENTAL PROCEDURE

MATERIALS

The materials used for this study were Alcoa MA-87 alloy and a 20 v/o SiCp-PM 7091Al MMC. The as-received MMC was cut from the as-produced extruded plate and had dimensions of 6.0 (15.3) x 5.0 (12.7) x 0.5 (1.27) inches (cm.). The production sequence of a typical SiC/Al alloy MMC has been described elsewhere (9). The typical compositions of a PM 7091Al pre-alloyed material, the as-received MMC and the matrix material equivalent MA-87 alloy are listed in Table 1. Chemical analysis was performed on a number of samples taken from the MMC plate using Inductively Coupled Plasma-atomic emission spectroscopy. Heat treatment times and temperatures used, for all specimens, are listed in Table 2. Density values were determined by dividing the MMC weight by its apparent volume. The calculations for determining the void content of the MMC were based on the as-produced theoretical density of 2.909 g/cm³, as given by the manufacturer. The density and percent void content values are listed in Table 3. The table also contains Rockwell B hardness values.

TABLE 1 - ELEMENTAL COMPOSITION OF MATERIALS

Material	Cu	Mn	Mg	Si	Cr	Fe	Zn	Ti	Co	Al
Prealloyed Powder (7091)	1.58	t	2.40	t	t	0.07	6.10	0.05	0.4	89.4
PM 7091/SiCp Composite	1.26	t	2.40	t	t	0.20	5.83	0.04	0.26	90.77
MA-87	1.50	—	2.50	—	0.10	0.10	6.50	—	0.40	89.9

t-Trace Quantities

TABLE 2 - HEAT TREATMENTS FOR PM SiCp/Al ALLOY MMC AND MA-87 Al ALLOY

Material Temper	Solution Heat Treatment	Aging
A	As received	—
B	910°F, 1 hr.	1. 4 days at room temp. 2. 24 hr. at 250°F
C	950°F, 2 hr.	1. 4 days at room temp. 2. 24 hr. at 250°F
MA-87	910°F, 2 hr. plus stretched	1. 24 hr. at 250°F 2. 4 hr. at 325°F

TABLE 3 - PHYSICAL PROPERTIES OF THE SiCp/Al ALLOY MMC

Material Temper	Rockwell B Hardness	Density g/cm ³	Void Content %
A	76	2.818 ± 0.082	3.13 ± 2.82
B	96	2.790 ± 0.045	4.09 ± 1.55
C	96	2.748 ± 0.083	5.53 ± 2.85

Theoretical plate density = 2.909 g/cm³Overall average density = 2.787 ± 0.076 g/cm³

Overall void content = 4.19 ± 2.61%

CORROSION STUDIES

The general corrosion behavior of the MMC and the MA-87 alloy, listed in Table 1, were determined by weight (mass) loss method (immersion tests) in 3.5% NaCl solutions of pH 2 and 6. All weight-loss test specimens were polished using standard metallographic techniques and finished to 1000 grit. Specimens [0.95(2.40)x 0.49(1.25)x 0.08(0.20) inch (cms.)] were exposed to the test solution for 1, 3, 10 and 15 days. After exposure, specimens were cleaned in 50 v/o nitric acid and dried before weighing. Also, solution pH was measured at each exposure interval. Exfoliation tests were performed on step specimens in accordance with the ASTM Standard Method G34-79 (14). The specimens were machined to expose the T/2, T/10(T=thickness of plate) and the top layer of the plate.

ELECTROCHEMICAL STUDIES

The electrochemical studies of the MMC and the MA-87 Al alloy were performed by measuring open circuit (corrosion) potentials, polarization resistances and potentiodynamic polarization (E vs. log i) behavior. Corrosion current densities, pitting potentials, and anodic and cathodic Tafel slopes were determined. The test solutions used were 3.5% NaCl, pH 2 and 6. The solution was deaerated with nitrogen gas one hour before and during each test. A saturated calomel reference electrode and a Pt counter electrode were employed. All specimens were cold-mounted in epoxy with their short transverse-longitudinal (S-L) face, 1 cm², open for exposure. The exposed metal surface was polished to a 1000 grit finish.

For the potentiodynamic polarization measurements, a potential range of -1.2 V to -0.6 V was selected with a scan rate of 0.166 mV/second. The potential was scanned from the cathodic to the anodic region. The potential scan was started as soon as the specimen was placed into the test solution. This procedure provided most reproducible results. Crevice and/or pit initiation during open-circuit potential-time measurements may have been the reasons for non-reproducibility in earlier studies (9). After testing, the specimens were examined microscopically. All electrochemical tests were performed with a PAR Model 351-2 Corrosion Measurement System.

RESULTS AND DISCUSSION

CORROSION STUDIES

Figure 1 shows plots of solution pH versus exposure time, and corrosion rate for each material. In both pH 2 and 6 solutions, the corrosion rates of MMC increased rapidly as the exposure time was increased to three days. Correspondingly, the pH of both the solutions also increased. A decrease in corrosion rate was observed for all specimens as the exposure time was increased to 15 days with only a slight increase in solution pH for each solution. The rate of increase of the solution pH appears to correlate with the change in corrosion rate. This indicates the pH sensitivity of the MMC's to corrosion. Overall, the temper A (as-received MMC) had the highest corrosion rate, followed by temper B then C. The MA-87 alloy had the lowest corrosion rate over a three-day exposure.

Test specimens were visually examined during the total immersion studies for preferential attacks, and corrosion products or deposition. After 15 days of exposure, in the pH 2 solution, material B specimens were covered with reddish-brown and some white corrosion products, temper C has a smaller amount of reddish-brown coverage while temper A specimens were coated with only white corrosion product. All specimens, during the 10 day exposure in pH 6 solution, were covered with a white corrosion product. The reddish-brown corrosion product was identified as mostly copper. For specimens tested in the pH 2 solution, the continuous corrosion and subsequent deposition of copper on the MMC surfaces was probably due to the corrosion of active intermetallics in Al alloy matrix material containing a large amount of copper (15). The modified solution heat treatment (for temper C) may have reduced the amount of active precipitates (e.g. $MgZn_2$) and intermetallics containing copper which forms during the aging process. Heat treatments may have caused a reduction in residual stresses at the SiC-matrix interfaces which would discourage solute (elemental) segregation or active phase precipitation. As a result less copper would be leached from temper C material.

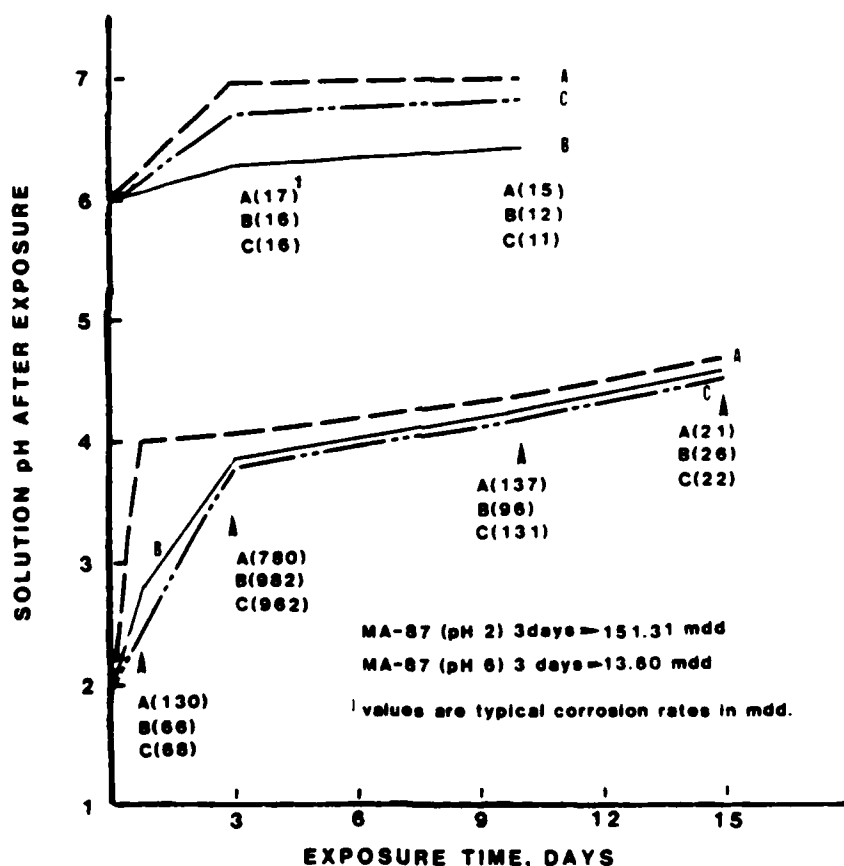


Figure 1. Effect of pH on Corrosion rates of the MMC and the MA-87 specimens with exposure time in pH 2 and 6, 3.5% NaCl.

Figure 2 shows photographs of the total immersion test specimens for the three and ten-day exposures. In the pH 2 test solution, all specimens were heavily corroded. Temper A showed some exfoliation; exfoliation corrosion may account for deviations in weight loss test results along with the loss of SiCp during exposure and specimen cleaning. Also, test solution or corrosion products lodged in deep pits or crevices could affect weight-loss test results. The specimens used for the pH 6 solution were not as badly corroded as those used in the pH 2 solution. In several cases, both pH 2 and 6 solutions, the corrosion attack of the MMC specimens was localized. This may be due to the loss of the protective oxide film on the Al alloy due to the SiC particles. Trzaskoma and McCafferty (1) found, for a SiCp/Al alloy MMC, the pit initiation usually occurs at SiCp clustered areas. The localized attack of the MMC specimens may be also due to processing; tempers B and C exhibited more localized attack than temper A. A lesser extent of localized attack for temper A, as compared to the SiCw/Al alloy MMC studied earlier (9), may reflect the lower void content, improved homogeneity of reinforcement phase and reduced elemental segregation of SiCp/Al alloy MMC. Chemical analysis tests did not show any significant deviation of constituent concentrations from sample to sample, although for one sample the Mg concentration was very low(0.40w/o).

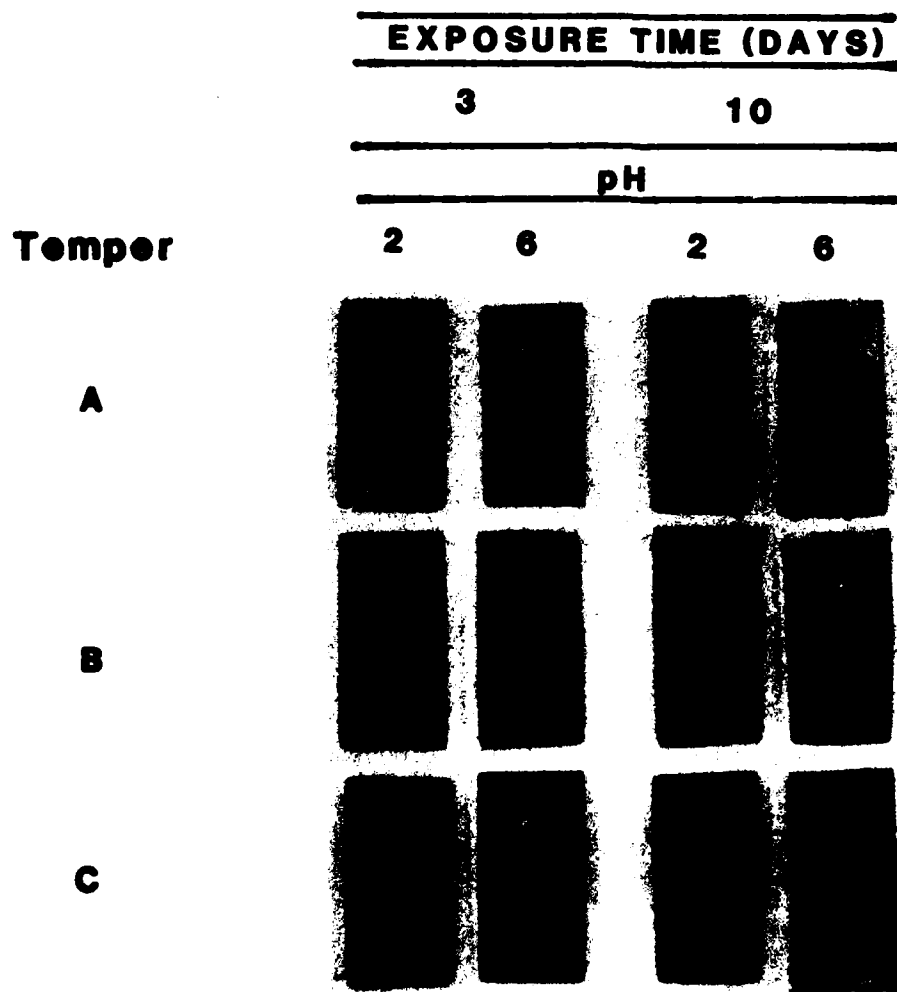


Figure 2. Weight-loss test specimens after exposure, in pH 2 and 6, 3.5% NaCl, for 3 and 10 days.

As shown in Table 3, heat treating increased the total void content of the MMC. Also, increasing the solution heat treatment time and temperature increased the void content of temper C as compared to temper B. Voids have been observed to expand during solution heat treatments of a PM 2024 Al alloy (16) and SiCw/Al alloy MMC (9). The larger void contents for material B and C may be due to thermal expansion and contraction of the SiCp reinforcement and/or growth of microvoids not eliminated during billet consolidation or during the extrusion process (17, 18). Figure 2 shows that tempers B and C have more areas of localized attack. The presence of voids in the MMC may be responsible for crevice or localized attack at the SiCp-matrix interfaces.

Figure 3 shows photographs of the exfoliation test specimens. As shown, tempers A, B and C have an EB, EC and EC EXCO rating, respectively. The MA-87 material shows pitting during the EXCO test (17). In the EXCO test, more corrosion products and copper deposits were found on temper C than B, and with a lesser amount on A. The reason for the severe attack of specimens B and C may be due to presence of active phases at the SiC-matrix interface. SiC particles acting as heat sinks during water quenching would significantly affect the solubility and diffusivity of solute elements in the matrix material, such that it could form areas containing only active components. The de-alloying and subsequential plating of copper could also be responsible for the severe attack observed for tempers B and C.

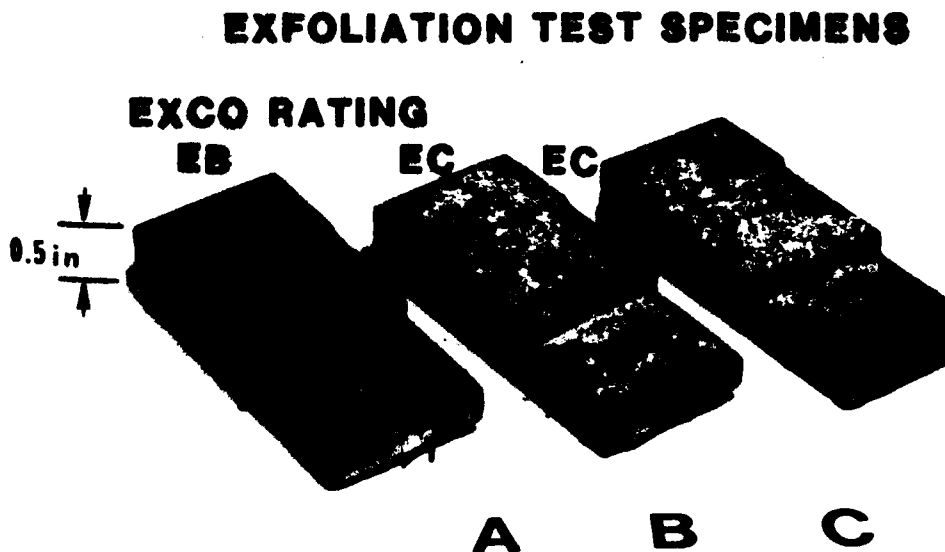


Figure 3. Exfoliation test specimens prior to removal of corrosion products.

Although the rolling faces of both the heat treated MMC specimens were severely exfoliated during the EXCO test, the S-L face remained only slightly corroded and did not show any type of preferential attack. As compared to SiCw/Al composite studied earlier (9), the corrosion resistance of the SiCp/Al alloy MMC, was better. The enhanced corrosion resistance of the S-L face for the SiCp/Al MMC may be due to the combination of the modified solution heat treatment and a higher mechanical deformation (extrusion ratio). Extruding the MMC billet at a higher extrusion ratio may have provided a more uniform substructure (13). This may have significantly affected subsequent precipitation reactions during aging at the SiCp-matrix interfaces and in the bulk of the MMC. The use of the SiCp instead of the SiCw gave a more uniform extrusion and improved homogeneity of the MMC billet.

Figure 4 shows micrographs of the S-L face for the MMC. As shown in Figure 4C, a high extrusion ratio produced a very uniform distribution of the SiCp; the Rockwell B hardness values measured were very uniform throughout the thickness of the plate. A hardness difference was observed through the thickness of the plate for SiCw/Al alloy MMC (9). As shown in Figures 4A, 4B, and 4D, the MMC does contain some SiCp clusters, SiCp and Al alloy free zones and stringers of the matrix material PM Al alloy. Thermal expansion of the SiCp in clustered areas could increase the local dislocation density in the matrix (18,19). The SiCp-matrix interfaces containing a high dislocation density are areas of high residual stresses. These areas could then become sites for crevice or pit formation (4).

ELECTROCHEMICAL STUDIES

The steady-state corrosion potentials for all materials tested are listed in Table 4 and 5. In the pH 2 solution, the corrosion potential of material temper A increased rapidly from -980 mV to -840 mV in approximately 500 seconds and then remained constant with time. This sharp rise to a steady-state potential may indicate homogeneity of the material. Similar trends were observed for the other materials. The initial potential after immersion was most noble for temper C and the most active for temper A. In all cases, except for A, the initial negative potentials may reflect the dissolution of active phases or intermetallics in the matrix material. Also, the steady-state corrosion potentials for materials B and C occurred at approximately 3000 seconds, about 2500 seconds later than for temper A. This slow rise to a steady-state potential for B and C is probably due to the presence of more active phases in the matrix metal. In pH 6, 3.5% NaCl solution, the initial corrosion potential-time response after total immersion was similar to the trend observed in pH 2 solutions. The most active to the least active initial potentials for the materials were the following order $A < B < MA-87 < C$. After approximately 2000 seconds all corrosion potentials became stable. The MA-87 alloy had the most active steady-state corrosion potential followed by temper C, B, and A. After exposure tests, the specimens were examined metallographically. Pits were observed to form around individual SiC particles and in the areas of SiC clusters.

Polarization resistance values for all materials tested are listed in Table 4 and 5. In pH 2 solution, the order of polarization resistance, R_p values was as follows: materials $C > B > MA-87 > A$. Thus, for the MMCs, temper A was active and had the lowest R_p . The lowest R_p for temper A may be due to corrosion occurring in crevices near SiCp-matrix interfaces, voids or in SiCp clustered areas. Since the polarization resistance measurement was performed after the steady-state potential was reached for each specimen, crevices may have formed earlier which may have reduced the values. In the pH 6 solution, the temper C showed the highest R_p value while temper A had the lowest.

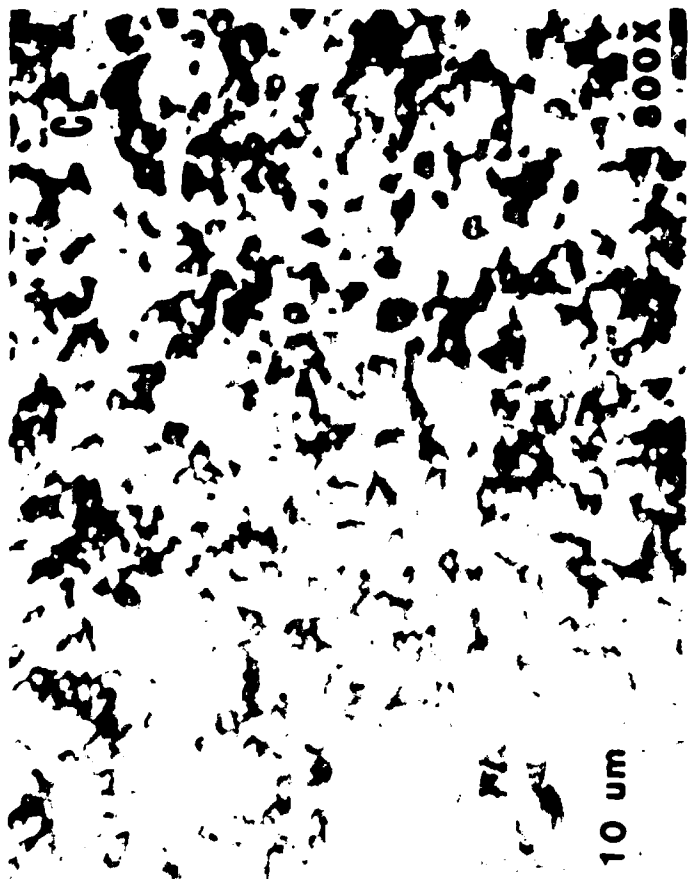


Figure 4 Optical micrographs of the S-L face of the SiCp. Al alloy MMC showing (A) and (B) Al alloy and SiCp free zones, (C) uniform SiCp distribution and (D) clustering of SiC particles

TABLE 4 - TYPICAL ELECTROCHEMICAL PARAMETERS OF MATERIALS IN 3.5% NaCl SOLUTIONS AT pH 2

Electrochemical Parameters	MMC Temper			MA-87
	A	B	C	
E_{oc}, mV	-831	-724	-724	-738
$R_p, \Omega \cdot cm^2$	2.62	12.8	22.8	10.0
$E(I=0), mV$	-840	-798	-798	-810
$\beta_c, mV/dec$	170	125	130	140
$\beta_a, mV/dec$	5	10	10	10
$i_{corr}, (\mu A/cm^2)$	630	250	200	100

TABLE 5 - TYPICAL ELECTROCHEMICAL PARAMETERS OF MATERIALS IN 3.5% NaCl SOLUTION

Electrochemical Parameters	MMC Temper			MA-87
	A	B	C	
E_{oc}, mV	-835	-845	-858	-880
$R_p, \Omega \cdot cm^2$	6.61E+3	7.51E+3	32.3E+3	11.5E + 3
$E(I=0), mV$	-926	-888	-819	-982
$R_p, \Omega \cdot cm^2$	34.9E+3	35.1E+3	63.4E+3	50.0E+3
$\beta_a, mV/dec$	133	82	80	90
$\beta_c, mV/dec$	144	163	226	129
E_{pit}, mV	-839	-816	-774	-765
$i_{corr}, A/cm^2$	9.0E-7	4.0E-7	2.0E-7	8.0E-7

The potentiodynamic polarization behavior for the MMC and the MA-87 alloy in pH 6 solution are given in Figure 5. Typical values of potentiodynamic parameters are summarized in Table 4 and 5 in pH 2 and 6 solutions, respectively. In pH 2 solution, MMC of tempers B and C have slightly lower corrosion current densities than for temper A. This order corresponds with that of the corrosion rates determined from the weight-loss measurements. In pH 6 solution, temper A exhibits a more active potential $E(I=0)$ and more negative pitting potential than for tempers B and C. Of the three MMC tempers, temper C shows the least negative $E(I=0)$ and pitting potential. These parameters are very significant and uniquely shown how modification in solution heat treatment can alter the corrosion behavior of a MMC. In pH 6 solution, MMC of temper C showed the lowest corrosion current density, followed by temper B and the MA-87 alloy. MMC of temper A had the highest corrosion current density. Contrary to the polarization behavior of the MMC, the MA-87 alloy showed a larger passive region.

The anodic Tafel slope for the MMC in the as-received condition (temper A) is greater than that for B and C in the pH 6 solution (Table 5). In the same solution, temper C had the greatest cathodic Tafel slope followed by temper B, A and the alloy MA-87. Greater the magnitude of the Tafel slope, the harder is the reduction of hydrogen. It has been reported earlier (9) that elemental segregation can occur during composite fabrication or heat treating and may cause a decrease in the cathodic Tafel slope. It was also observed, for SiC/Al MMC, that copper can segregate and shift the Tafel slope to higher values (9). This trend was not observed on the SiCp/Al alloy MMC, thus, may be considered a homogeneous (structure) material.

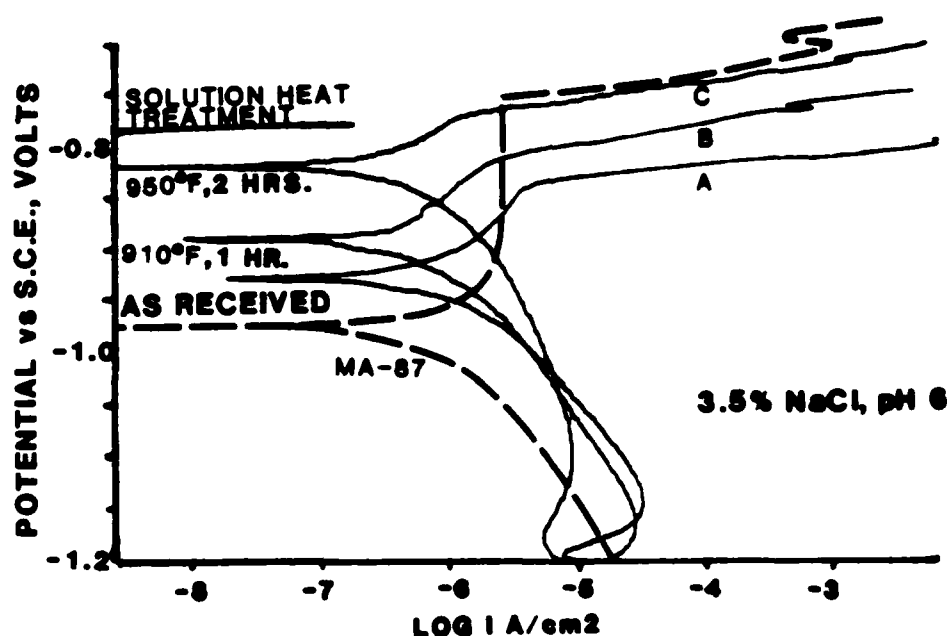


Figure 5. Potentiodynamic polarization behavior of the MMC and the MA-87 Al alloy specimens.

CONCLUSIONS

The effects of fabrication and processing on the corrosion susceptibility of a PM 7091Al alloy-20 v/o SiCp composite was studied. It was found that the MMC did not exhibit any abnormal corrosion behavior as was observed for SiCw/Al MMC, described elsewhere (9). Although the SiCp distribution was very uniform, the composite did have some clustered SiCp. Clustered areas of SiC particles were regions of enhanced corrosion attack. The MMC's corrosion resistance was improved by a combined effect of a modified solution heat treatment and a high extrusion ratio (higher mechanical deformation). Solution heat treatment of the as-received material caused an increase in the void content of the MMC which may have resulted in a decrease in corrosion resistance (exfoliation and general corrosion). Expansion and contraction of the SiC particles may cause a high dislocation density near the SiCp-matrix interface during heat treating and water quenching (19), and may have increased the amount of active phase precipitating during subsequent aging (18).

Generally, it has been concluded that processing variables play an important role in the corrosion behavior of MMC, and that a high extrusion ratio and a modified solution heat treatment can enhance the corrosion resistance significantly.

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